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Evaporation Temperature Alters Physicochemical Characteristics and Volatile Maillard Reaction Products of Non-Centrifugal Cane Sugar (NCS): Comparison of Polyethylene Membrane and Retronasal Aroma Simulator Techniques for the Extraction of Volatile Organic Compounds in NCS

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Featured Application: This study shows the impact of altering the evaporation temperature during the production of non-centrifugal cane sugar (NCS) on its physicochemical and flavor qualities. Temperature adjustment during the evaporation process can be a key factor in determining the moisture content, water activity, color, and aroma of the NCS. This information can be used as a basis for predicting the storage stability and sensory characteristics of NCS.

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Abstract: Non-centrifugal cane sugar (NCS) is produced from sugarcane syrup via thermal evaporation. This study aimed to assess the effects of different temperatures during the evaporation process on the physicochemical characteristics and Maillard reaction products (MRPs) of NCS. Evaporation was tested at three final heating temperatures (120, 130, and 140 °C). The moisture content, water activity, L*a*b* color spaces, and ICUMSA (International Commission for Uniform Methods of Sugar Analysis) values of the NCS were determined. Volatile MRPs of NCS were extracted using polyethylene (PE) membrane and retronasal aroma simulator (RAS) techniques, and their components were measured using gas chromatography. A higher evaporation temperature produced NCS with less moisture content and water activity. However, it also led to a darker color, as indicated by lower L* (brightness) and b* (yellow) values in the color spaces. Additionally, higher evaporation temperatures resulted in greater ICUMSA values. Moreover, higher heating increased the amounts of volatile MRPs, such as 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one, 2-furanmethanol, 2-methylpyrazine, 2,5-dimethylpyrazine, and 2,6-dimethylpyrazine. Negative correlations were observed between moisture content, water activity, brightness, yellow color, and the total MRPs obtained by PE and RAS extractions. Additionally, positive and significant correlations were confirmed between ICUMSA values and most MRPs. Thus, the evaporation temperature alters the key physicochemical traits and volatile compounds of NCS, affecting its physical stability and flavor quality.

Keywords: non-centrifugal cane sugar; evaporation temperature; physicochemical traits; Maillard reaction products; polyethylene membrane extraction; retronasal aroma simulator

1. Introduction

Non-centrifugal cane sugar (NCS) is an unrefined, solidified sugar produced from sugarcane syrup without molasses removal. The non-centrifugation evaporation process

includes extensive syrup heating and drying–solidification steps with continuous agitation [1]. The thermal process, especially the final heating stage at temperatures between 100–140 °C, facilitates a non-enzymatic browning mechanism, known as the Maillard reaction, in the solidified mixture [1–3]. This browning reaction occurs between carbonyl compounds (reducing sugars) and amino compounds (amino acids) in heated sugarcane syrups [3,4]. The manufacturing method is also responsible for the increased concentration of biofunctional components, such as minerals, polyphenols, and policosanols, in NCS than in refined sugar [1,5,6]. In Japan, NCS is known as kokuto or kurozato, which means brown or black sugar. NCS has been an important sugary delicacy in Okinawa Prefecture, where sugarcane plantations occupy approximately 50% of its farmland. The sugarcane industry accounts for 20% of the gross agricultural production of the prefecture; thus, NCS plays an important role in supporting its society and economy [7].

The physicochemical characteristics and volatile compounds in food play critical roles in determining sensory preferences and consumer acceptance [2,8]. These factors can significantly affect the overall quality and taste of food products, including NCS [5,8]. Physicochemical traits such as moisture content and water activity may predict the shelf-life stability and storage conditions of a product [3,9]. Meanwhile, the brown color, which is a key element in the visual appearance of NCS, may influence its sensory perceptions and practical applications. It has been found to be positively associated with the accumulation of bioactive substances in NCS [1,2,8]. Among the volatile compounds, Maillard reaction products (MRPs), such as pyrazines, furans, pyranones, and pyrroles, are considered key flavor components of NCS that generate various desirable odors [5,8,10]. These volatile MRPs emit nutty, buttery, roasted, toasted, and sweet caramel aromas, which contribute to their sensory properties [1,2,10].

The volatile components of foods can be isolated using various extraction methods, including solvent extraction, solid-phase microextraction, and headspace extraction techniques [6,10,11]. Each method enables the isolation of numerous volatile substances from different chemical groups. However, complete extraction of whole volatile compounds is difficult to achieve using a single extraction technique [6,11,12]. Nevertheless, the above methods were tested on NCS products in our previous studies and provided important information on the comparable composition and key active aromatic compounds of NCS [1,3,5]. Polymer membrane-assisted extraction via polyethylene (PE) films, such as low-density polyethylene (LDPE), allows the permeation of low-molecular-weight volatile substances through the non-crystalline structures of the membrane barrier [13]. On the other hand, most non-volatile components cannot penetrate the barrier, leading to the separation of volatile compounds [13,14]. A thin PE film can easily be folded to form a pouch shape for the extraction of volatile components. This is an effective interface for isolating volatile substances of different chemical polarities and matrices from aqueous food materials, such as butter, oil, and soy sauce, to organic solvents on the outer side of the film membrane. As a result, it offers a simple technique for volatile extraction [13,15]. Furthermore, retronasal aroma, which represents the odor released from foods during chewing or drinking, has recently garnered much interest in flavor studies [16,17]. The retronasal aroma simulator (RAS) isolates volatile compounds released from food matrices in reaction with artificial saliva, which mimics the human oral cavity and olfactory systems [17,18].

There is limited information on the effects of thermal processes on the physicochemical and flavor qualities of NCS products. Therefore, the current study aimed to assess the impact of different evaporation temperatures during the final heating stage on the physicochemical characteristics and volatile MRPs of NCS. The NCS was produced using tabletop-type manufacturing equipment [19], with the maximum evaporation temperature of the final heating process at 120, 130, or 140 °C. The physicochemical traits of the NCS were evaluated, including moisture content, water activity, and color indicators (L*a*b* color spaces and International Commission for Uniform Methods of Sugar Analysis [ICUMSA] values). To the best of our knowledge, this is the first study to attempt to isolate volatile MRPs from NCS using PE membrane and RAS extraction techniques. Both

extraction techniques were applied to monitor changes in the composition and amount of volatile MRPs in NCS products at different evaporation temperatures. Moreover, the RAS extraction technique was applied to present an in vitro retronasal aroma model of NCS.

2. Materials and Methods

2.1. Reagents

Cyclohexanol, $NaHCO_3$, and K_2HPO_4 were purchased from Fujifilm Wako Pure Chemical Industries (Osaka, Japan). $Ca(OH)_2$ and NaCl were purchased from Kanto Chemical Industry (Tokyo, Japan), and $CaCl_2$ was obtained from Tokyo Chemical Industry (Tokyo, Japan). KCl was purchased from Nacalai Tesque (Kyoto, Japan), and α-amylase was obtained from Sigma-Aldrich (St. Louis, MO, USA). Authentic standards for the identification of volatile components were purchased from Sigma-Aldrich and the Tokyo Chemical Industry. All the other reagents were of analytical grade.

2.2. NCS Production

Sugarcane stalks of the "Nourin No. 15" cultivar were harvested at the mature stage in December 2019 from a farm at the Okinawa Prefectural Agricultural Research Center, Okinawa, Japan. The sugarcane stalks were crushed using a roller-type crusher, and the squashed juice (Brix 23.9%, pH 5.17) was purified using cold liming through calcium hydroxide addition. The mixture was centrifuged at 6000 rpm for 20 min, and the clarified juice (supernatant) was heated to prepare sugarcane syrup (50% Brix). The NCS was produced from 800 g of syrup using tabletop-type manufacturing equipment (Nishikawa Keisoku, Tokyo, Japan) [19]. The syrup was processed in two thermal evaporation stages: final heating and solidification (Table 1). Each stage comprised four heating segments. The incremental temperatures of the final heating stage were tested in three patterns, in which the syrup temperature at heating segment No. 4 was set at 120, 130, or 140 °C. Changes in the syrup temperature during production were recorded, as shown in Figure 1a. The rotation of the mixing blade was set to 100 rpm and 200 rpm in the final heating and solidification stages, respectively. The final torque of the rotor during the solidification evaporation was set to 2.08 N·m. Solidified NCS (approximately 300 g) was collected from the manufacturing equipment after finishing the thermal process of heating segment 8, and the solidified product was immediately crushed into a powder (Figure 1b). All experiments were performed in triplicate. The powdered NCS was stored at −30 °C prior to analysis.

Table 1. Evaporation (final heating and solidification) temperature settings in non-centrifugal cane sugar (NCS) production.

Parameter	Final Heating Temperature (°C)				Solidification Temperature (°C)			
Heating segment No.	1	2	3	4	5	6	7	8
Heater temperature	250	220	220	220	140	140	140	140
Syrup temperature of pattern 120 °C [1]	105	110	115	120	115	110	105	80
Syrup temperature of pattern 130 °C [1]	115	120	125	130	115	110	105	80
Syrup temperature of pattern 140 °C [1]	125	130	135	140	115	110	105	80

[1] Set temperature on syrup; the maximum temperatures of syrup at heating segment No. 4 were set at 120, 130, and 140 °C. To achieve the maximum temperatures, different stepwise set temperature increments at the previous heating segments (segments No. 1–3) were applied.

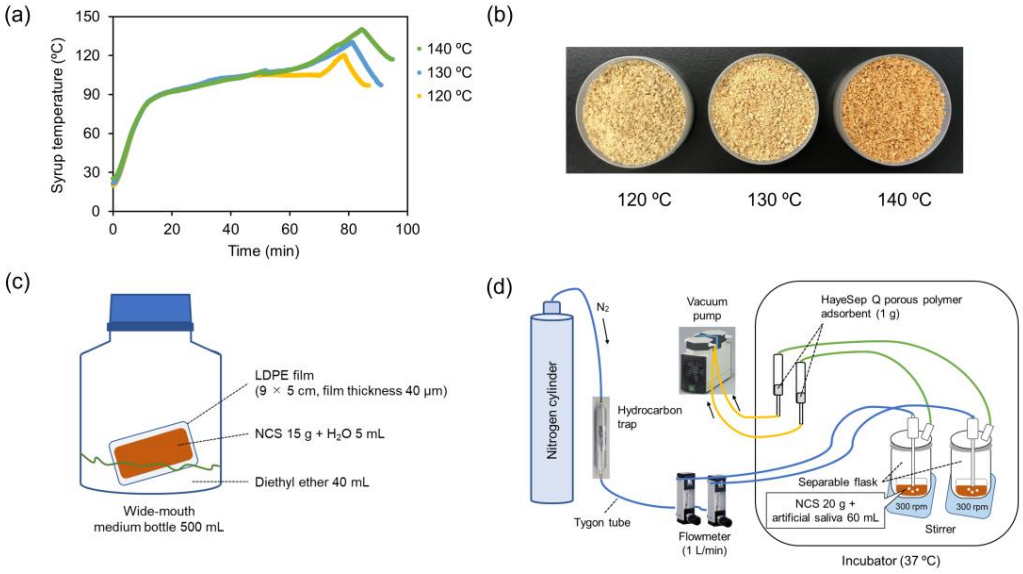

Figure 1. (**a**) Typical temperature changes during non-centrifugal cane sugar (NCS) production at different evaporation temperatures; (**b**) photograph of powdered NCS; (**c**) polyethylene (PE) membrane extraction scheme; (**d**) retronasal aroma simulator (RAS) extraction scheme.

2.3. Measurements of Physicochemical Characteristics

The moisture content and water activity of the NCS were measured using moisture content (MOC63u, Shimadzu Corporation, Kyoto, Japan) and water activity analyzers (LabSwift-aw, Novasina, Lachen, Switzerland), respectively [1]. The L*a*b* color spaces of the NCS solution (20%, w/v) were determined using CM-2006d in specular component-included mode (Konica Minolta, Tokyo, Japan) [8]. The NCS color was evaluated according to the ICUMSA GS1/3e7 protocol [20] using a spectrophotometer (SH-9000Lab, Corona Electric, Ibaraki, Japan). All assays were performed in triplicate.

2.4. PE Membrane Extraction of Volatile MRPs

The MRPs of the NCS were isolated through the PE membrane (Figure 1c). Briefly, NCS (15 g), Milli-Q water (5 mL), and the internal standard cyclohexanol (0.1%, w/v, 10 μL) were placed in a three-side-sealed LDPE pouch (9 × 5 cm, film thickness 40 μm, Unipac C-4, Seisannipponsha, Tokyo, Japan). The pouch was then sealed on the upper side [15]. Subsequently, the pouch and 40 mL of diethyl ether were placed in a 500 mL wide-mouth medium bottle, and the bottle was incubated in a water bath for 3 h at 30 °C with constant shaking (70 rpm). Afterward, the pouch was removed from the bottle, and the extract was dehydrated with 2 g anhydrous sodium sulfate for 12 h at 5 °C. The volume was then reduced to 50 μL using a Kuderna–Danish concentrator tube under a gentle nitrogen stream (99.99% purity, Okano, Naha, Japan). The volatile MRP extract was stored at −30 °C prior to analysis. All extractions were performed in triplicate.

2.5. RAS Extraction

MRPs of NCS were isolated using the RAS system at 37 °C for 1 h in an FMU-0541 incubator (Fukushima Kogyo, Osaka, Japan) (Figure 1d). The apparatus in the system was connected through Tygon tubes (7.5 mm id, Nihon Pisco, Nagano, Japan). Briefly, NCS (20 g), artificial saliva (60 mL), internal standard cyclohexanol (0.1%, w/v, 50 μL), and a 2.5 cm-PTFE magnetic stirring bar were placed in a crystallizing dish (7 × 5 cm). The artificial saliva was composed of $NaHCO_3$ (62 mM), NaCl (15 mM), KCl (6.4 mM), K_2HPO_4 (6 mM), $CaCl_2$ (3 mM), and α-amylase (5 mg) [18]. The dish was placed in a 1 L separable flask outfitted with a two-neck cover, and the flask containing the mixture solution was then mixed using a magnetic stirrer at 300 rpm. A constant flow of nitrogen was streamed at 1 L/min to the mixture, and the released volatiles were absorbed into 1 g of

the HayeSep Q porous polymer (80–100 mesh, Supelco, Bellefonte, PA, USA) under vacuum suction using an EVP-1100 vacuum pump (Tokyo Rikakikai, Tokyo, Japan). Afterward, the volatiles were diluted with diethyl ether (20 mL), and the extract was dehydrated with 2 g anhydrous sodium sulfate for 12 h at 5 °C. The volume was then reduced to 50 μL using a Kuderna–Danish concentrator tube under a gentle nitrogen stream. The volatile MRP extract was stored at −30 °C prior to analysis. All extractions were performed in triplicate.

2.6. Gas Chromatography (GC) Analysis

The MRPs of the concentrated volatile extracts were analyzed using GC techniques [1], namely GC–FID/MS (flame ionization detection/mass spectrometry) analysis for PE membrane extract and GC–MS analysis for RAS extract. The GC–FID was performed using a 6890N GC equipped with a DB-WAX column (60 m × 0.25 mm i.d., film thickness 0.25 μm) (Agilent Technologies, Santa Clara, CA, USA). The PE membrane extract (1 μL) was injected at a split ratio of 1:10. The temperatures of both the GC injector and detector were set at 250 °C. The column temperature was initially set at 40 °C for 1 min, raised to 200 °C at a rate of 3 °C/min, and then maintained at that temperature for 37 min. The helium carrier gas was set to a linear velocity of 32 cm/s. Subsequently, compound identification was performed using a 7890A GC-5975C MS (Agilent Technologies) under the same GC conditions described above. For MS detection, the ion source and interface were maintained at 230 °C. The ionization energy was 70 eV, and the mass acquisition range was *m/z* 33–350.

The RAS extract was analyzed using a 7890B GC-5977A MS equipped with a DB-WAX column (30 m × 0.25 mm i.d., 0.25 μm) (Agilent Technologies). The column was initially set at 40 °C for 1 min, raised to 200 °C at a rate of 3 °C/min, and maintained at this temperature for 17 min. The other GC parameters and MS conditions were set as described above.

Compounds were identified by comparing linear retention indices (RIs) using a homologous series of *n*-alkanes (C_7–C_{30}) and mass spectral fragmentation patterns with MS data from the National Institute of Standards and Technology MS Library (Version 2008 or 2014). Co-injected authentic standards were also used for confirmation of compound identification. The weight intensity of the identified peak was calibrated to the FID or MS response of the internal standard, and the compound content was expressed as μg/100 g. All assays were performed in triplicate.

2.7. Statistical Analysis

The average and standard deviation of the physicochemical properties and aroma components were calculated. The significant differences among the groups were analyzed using the Tukey–Kramer HSD test (JMP Version 13, SAS Institute, Cary, NC, USA). Pearson's correlation analysis was performed using GraphPad Prism Version 9 (GraphPad Software, La Jolla, CA, USA).

3. Results

3.1. Physicochemical Characteristics of NCS

Evaporation temperature greatly altered the physicochemical traits of the NCS, including moisture content, water activity, and color indicators (Table 2). The moisture contents of NCS products obtained with final heating temperatures of 120, 130, and 140 °C were 5.98, 4.41, and 2.72%, respectively. The water activity of the NCS also significantly decreased by nearly half, from 0.63 to 0.33, as the temperature increased from 120 to 140 °C ($p < 0.05$). Both the L* (brightness) and b* (yellow) color spaces declined significantly from 34.23 to 26.44 and 19.03 to 3.90, respectively. However, the intensity of the red color indicator (a* color space) varied across the three temperature levels, with the highest intensity (12.95) observed in the NCS of the final heating temperature at 130 °C. The image presentation of the combination of the three-color spaces confirmed the effect of the enhanced evaporation temperature on the dark brown color of the NCS. Moreover, the ICUMSA values of NCS significantly increased from 16,005 to 36,188 IU as the evaporation temperature was raised from 120 to 140 °C.

Table 2. Moisture content, water activity, L*a*b* color spaces, and ICUMSA values of non-centrifugal cane sugar (NCS) were obtained with different evaporation temperatures.

Trait	120 °C	130 °C	140 °C
Moisture content (%)	5.98 ± 0.24 a [1]	4.41 ± 0.18 b	2.72 ± 0.17 c
Water activity	0.63 ± 0.01 a	0.55 ± 0.03 b	0.33 ± 0.04 c
L*	34.23 ± 2.23 a	32.48 ± 0.43 b	26.44 ± 0.76 c
a*	10.34 ± 0.41 b	12.95 ± 0.27 a	7.98 ± 2.02 c
b*	19.03 ± 0.66 a	13.97 ± 0.67 b	3.90 ± 1.26 c
L*a*b* color spaces image presentation [2]			
ICUMSA value (IU)	16,005 ± 488 c	19,648 ± 142 b	36,188 ± 4133 a

[1] Each value is expressed as the mean ± standard deviation (n = 3). Different letters in the same row indicate significant differences at $p < 0.05$. [2] Generated by http://colorizer.org (accessed on 1 December 2020).

3.2. Volatile MRPs of NCS

The evaporation temperature affected the MRP composition of the NCS. The volatile extract of the PE membrane extraction contained seven compounds (Figure 2), namely three pyrazines, two furans, one pyranone, and one pyrrole, and their amounts varied in the NCS products. The most dominant MRP was 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one, and the amount of this compound significantly increased from 378.12 to 3821.94 μg/100 g ($p < 0.05$) as the evaporation temperature was raised from 120 to 140 °C (Figure 2a). The second predominant compound, 2-furanmethanol, was found in much higher concentration in the NCS obtained with a final heating temperature of 140 °C than in the two other NCS products (432.46 vs. 31.20–88.89 μg/100 g, respectively) (Figure 2b). Moreover, except for 2-acetylpyrrole, significant incremental trends were observed in the other MRPs with increasing temperature (Figure 2c–g). The total amounts of MRPs in the NCS products from final heating temperatures at 120, 130, and 140 °C were 448.84, 1550.09, and 4386.98 μg/100 g, respectively. There was a 2.45-fold increase in total volatile MRPs by increasing the evaporation temperature from 120 to 130 °C and a 1.83-fold increase when the temperature was raised from 130 to 140 °C (Figure 2h).

The RAS extraction system isolated four volatile MRPs from the NCS: 2-furanmethanol, 2-methylpyrazine, 2,5-dimethylpyrazine, and 2,6-dimethylpyrazine (Figure 3a–d). Notably, 2-furanmethanol was absent in the NCS obtained at the evaporation temperature of 120 °C, but its amount was greatly enhanced from 11.92 to 113.05 μg/100 g in the NCS when the evaporation temperature was raised from 130 to 140 °C (Figure 3a). The pyrazine concentrations ranged from 12.09–155.44, 18.32–41.06, and 17.41–55.15 μg/100 g for 2-methylpyrazine, 2,5-dimethylpyrazine, and 2,6-dimethylpyrazine, respectively. These concentrations were significantly enhanced with an increase in the evaporation temperature from 120 to 140 °C (Figure 3b–d). The total MRPs in the NCS ranged from 47.81 to 364.69 μg/100 g, wherein the MRP concentration increased by 1.52- and 2.03-fold with an increase in the temperature from 120 to 130 °C and 130 to 140 °C, respectively (Figure 3e).

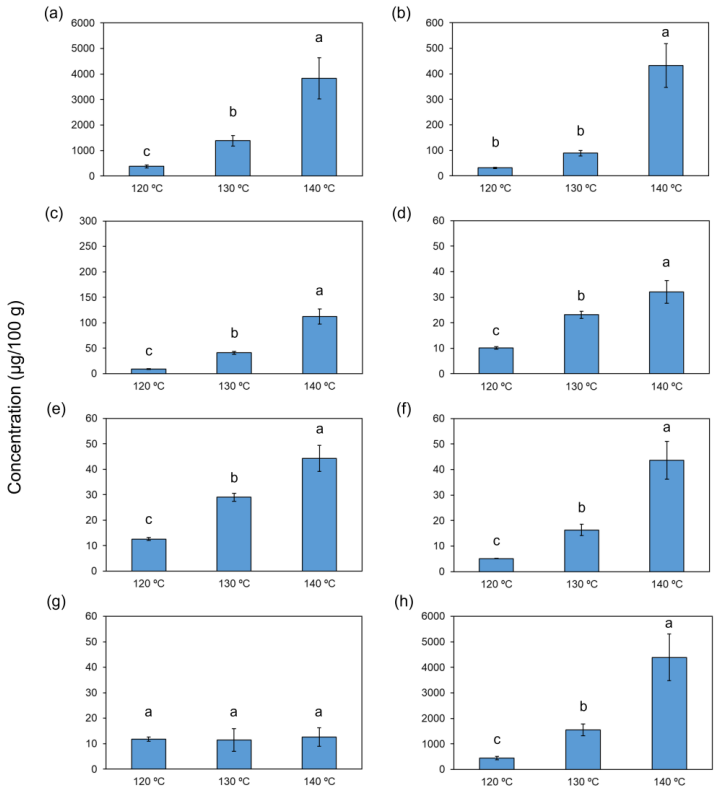

Figure 2. Maillard reaction products (MRPs) of non-centrifugal cane sugar (NCS) obtained at different evaporation temperatures by PE membrane extraction: (**a**) 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one; (**b**) 2-furanmethanol; (**c**) 2-methylpyrazine; (**d**) 2,5-dimethylpyrazine; (**e**) 2,6-dimethylpyrazine; (**f**) 5-methyl-2-furanmethanol; (**g**) 2-acetylpyrrole; (**h**) Total MRPs. Each value is expressed as the mean ± standard deviation (n = 3). Different letters indicate significant differences at $p < 0.05$.

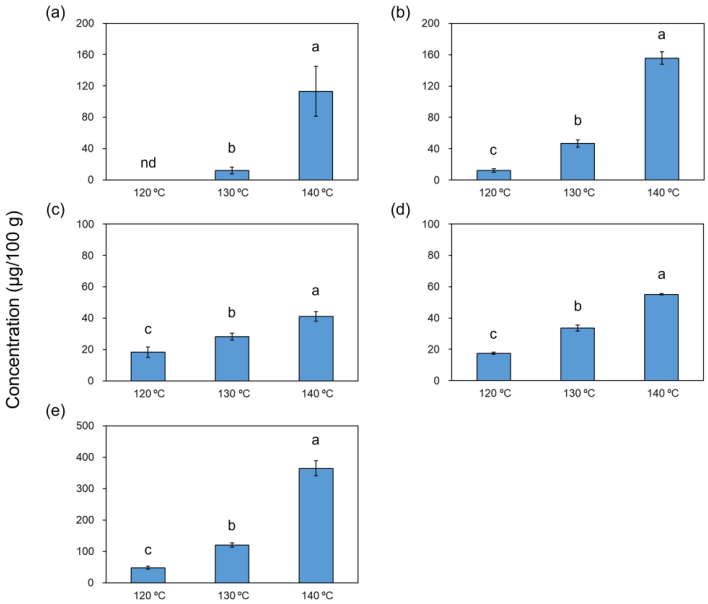

Figure 3. Maillard reaction products (MRPs) of non-centrifugal cane sugar (NCS) obtained at different evaporation temperatures by RAS extraction: (**a**); 2-furanmethanol (**b**); 2-methylpyrazine (**c**); 2,5-dimethylpyrazine (**d**); 2,6-dimethylpyrazine (**e**); total MRPs. Each value is expressed as the mean ± standard deviation (n = 3); nd: not detected. Different letters indicate significant differences at $p < 0.05$.

3.3. Correlations between Physicochemical Characteristics and Volalite MRPs of NCS

Pearson's correlations between the physicochemical traits, MRPs of the PE extraction, and MRPs of the RAS extraction were visualized using a heatmap (Figure 4). The moisture content of the NCS obtained at different evaporation temperatures was positively related to water activity, brightness (color space L*), and yellow color (color space b*). This relationship was supported by high Pearson's correlation coefficients of 0.963, 0.883, and 0.980, respectively. These associations were found to be statistically significant at $p < 0.01$. Water activity was also significantly correlated with brightness and yellow color. Negative correlations were observed between these two physical traits and the most volatile MRPs in the NCS (except for 2-acetylpyrrole). However, significant positive correlations were confirmed between ICUMSA values and these MRPs ($p < 0.01$). Furthermore, a strong association was observed between the total MRPs from the PE and RAS extractions, with a Pearson coefficient of 0.948. Additionally, there were significant positive correlations between the amounts of 2-furanmethanol, 2-methylpyrazine, 2,5-dimethylpyrazine, and 2,6-dimethylpyrazine compounds obtained using the two extraction systems ($p < 0.01$).

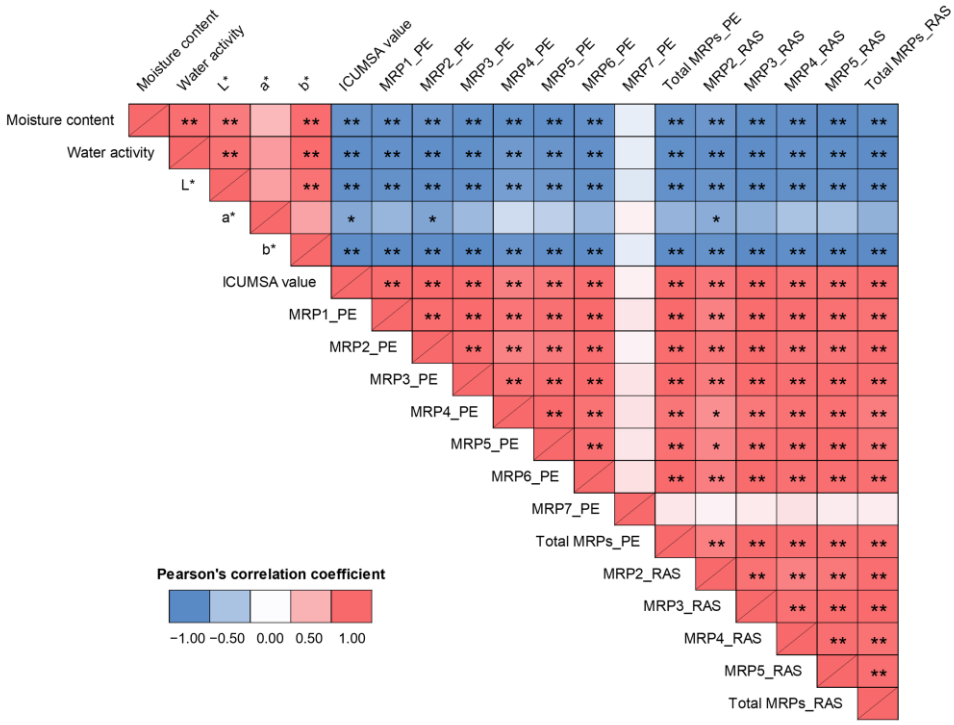

Figure 4. Heatmap visualization of Pearson's correlation between physicochemical characteristics and Maillard reaction products (MRPs) of non-centrifugal cane sugar (NCS) obtained at different evaporation temperatures. Asterisks indicate a significant correlation; * $p < 0.05$; ** $p < 0.01$. MRP1: 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one; MRP2: 2-furanmethanol; MRP3: 2-methylpyrazine; MRP4: 2.5-dimethylpyrazine; MRP5: 2.6-dimethylpyrazine; MRP6: 5-methyl-2-furanmethanol; MRP7: 2-acetylpyrrole; PE: polyethylene membrane extraction; RAS: retronasal aroma simulator extraction.

4. Discussion

Physicochemical characteristics such as moisture content, water activity, and color are important for NCS because these traits influence consumer sensory preferences [8,21]. The moisture content of the NCS products obtained at different evaporation temperatures was below 6% (Table 2). These values fall within a similar range as those reported for commercial unrefined sugars in previous studies [1,22]. The water activity of NCS produced with an evaporation temperature of 140 °C was markedly below 0.4, suggesting potent long-term storage stability, equivalent to dried foods [9,23]. Moreover, the water activity level of

NCS obtained at the lowest evaporation temperature of 120 °C was 0.63, which can be considered an adverse condition for the growth of spoilage microbes [9,24]. Therefore, this thermal process was adequate to preserve the product against potential microbial spoilage. The values of these physical traits decreased as the evaporation temperature increased, indicating the importance of the thermal process in NCS production. This thermal process plays a crucial role in evaporating moisture from the solidified products, confirming the widely accepted notion that thermal energy influences the mass balance in the evaporation process [25,26].

Temperature adjustments during the evaporation process also affected the color of the NCS; the solidified products became darker as the temperature increased, as evident from the visual and color indicators (Figure 1b; Table 2). This color development could be caused by the accumulation of brown pigment substances in the NCS, which are formed through Maillard reactions and caramelization during heating [2,3,27]. The color indicator data, which are numerical representations of color properties such as L*a*b* color spaces and ICUMSA values, are very important for the evaluation of NCS. By measuring these indicators, manufacturers can ensure that they consistently meet the desired color specifications, which are important for the appearance, acceptance, and marketability of these products [2,3,22]. These physicochemical data can also be used to monitor the effects of processing, storage, and other factors on the color of NCS and their derived food and beverage products. This enables manufacturers to make the necessary adjustments to ensure that the final products meet their desired quality standards [3,19,21].

The Maillard reaction, which occurs rapidly during the final heating stage of the evaporation process, is closely related to the unique color, taste, and aroma of NCS [8,10,22]. The rate of the Maillard reaction is temperature-dependent. An increase in temperature can enhance the reaction rate because it influences the activation energy required for the reaction to occur [28,29]. At higher temperatures, the precursor molecules (reducing sugars and amino acids) have greater kinetic energies, which enables them to overcome the activation energy barrier and react more rapidly [29,30]. As a result, the Maillard reaction can occur more quickly, leading to a higher formation of MRPs. This phenomenon can account for the increased generation of aroma compounds, such as pyranone, furan, pyrazine, and pyrrole (Figures 2 and 3), in the NCS obtained with high evaporation temperatures. Moreover, the PE and RAS extraction systems showed different compositions of volatile MRPs. Specifically, the RAS extract was found to lack the presence of 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one, 5-methyl-2-furanmethanol, and 2-acetylpyrrole. The lack of pyranone in the RAS extract is probably due to its high boiling point, which might impact the volatilization of the substance during extraction compared to pyrazines (281 vs. 135–155 °C, respectively). RAS also has limitations in isolating minor components such as 5-methyl-2-furanmethanol and 2-acetylpyrrole, indicating its limited capability to extract large volatiles. Nevertheless, the RAS system can effectively display changes in the key volatiles of the evaluated materials [16–18]. The results indicate that as the evaporation temperature increases, the following odors in NCS may potentially intensify due to the presence of MRPs: sweet-caramel and maple (2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one); roasted-nutty and fruity (2-furanmethanol); sweet and minty (5-methyl-2-furanmethanol); sweet and grassy (2-methylpyrazine); roasted-nutty and earthy (2,5-dimethylpyrazine); and sweet and nutty (2,6-dimethylpyrazine) [1,10]. This variation offers a wide range of NCS products with different potent flavor properties for table snack consumption or as raw materials for food and beverage production.

The statistical correlations depicted in Figure 4 measured the degree of linear association between moisture content, water activity, L*a*b* color spaces, ICUMSA values, and MRPs in the NCS. The high correlations between these variables showed that NCS from higher evaporation temperatures tended to have a darker appearance, were more preservable, and contained more volatile MRPs. These MRPs were responsible for emitting potent aromas such as sweet caramel and roasted peanuts [10]. Through the analysis of the correlation between physicochemical traits and volatile MRP levels, NCS manufac-

turers can enhance their understanding of the chemical reactions that take place during processing. This knowledge enables them to identify potential strategies for controlling and optimizing the production of desirable aroma compounds, such as volatile MRPs, through thermal process adjustment [8,19]. Strong statistical correlations also have important implications for quality control and energy use for evaporation during production. As a result, they can be used to improve both the quality of the NCS and the energy efficiency of the process [1,28,30].

The outcomes of this study have broader applications beyond characterizing the physical traits, color indicators, and composition of volatile compounds in freshly produced NCS. They can also be used to predict the consistency and quality of NCS products during storage and estimate their potent flavor quality when they are applied to foods and beverages [8,21]. Taken together, the thermal evaporation process in NCS production affected its physicochemical characteristics and volatile MRPs, and thus, its quality. This information is valuable for understanding and modifying key parameters in NCS production, particularly when optimizing energy costs in large-scale manufacturing. It also helps in achieving optimal manufacturing parameters to produce NCS with the desired storage stability and flavor quality required for various food and beverage applications.

5. Conclusions

The final heating temperature in the evaporation process during NCS production, which operates between 120 and 140 °C, clearly altered its physical characteristics and volatile MRPs. Moisture content and water activity were reduced by half in the NCS from 140 °C heating compared to those in the NCS obtained from 120 °C evaporation. Increased evaporation temperature also produced NCS with a less bright and yellow color, as well as a greater ICUMSA value (darker color), which greatly contributed to its appearance and color. The final heating temperature was also responsible for variations in the generation of volatile MRPs, including pyranone, furan, and pyrazine compounds. Higher evaporation temperatures increased the amounts of most of the MRPs isolated by PE membrane extraction, except for 2-acetylpyrrole. Moreover, the RAS system confirmed the presence of four MRPs: 2-furanmethanol, 2-methylpyrazine, 2,5-dimethylpyrazine, and 2,6-dimethylpyrazine. The concentrations of these compounds showed an increase with higher evaporation temperatures. There were strong negative correlations between physicochemical traits such as moisture content, water activity, brightness, yellow color, and MRPs from PE and RAS extractions. Conversely, significant positive correlations were confirmed between ICUMSA values and the most volatile MRPs. Regarding volatile MRPs' isolation, the current study has focused on PE and RAS extraction techniques, wherein the isolated odorants may differ from those that human olfactory receptors can perceive. Nevertheless, these volatile extraction techniques effectively displayed the increments of volatile MRPs over evaporation temperature changes. Notably, the RAS presented for the first time an in vitro retronasal aroma model for NCS. Therefore, confirmation of the relationship between retronasal olfaction and sensory profiles could be an attractive topic for future research on NCS flavor quality. Taken together, the study shows that the evaporation temperature plays a crucial role in altering the key physicochemical and flavor characteristics of NCS. This outcome highlights the importance of adjusting the evaporation temperature during NCS production to improve storage stability and sensory characteristics, and thus quality.

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